# Mechanical Properties of Hot Pressed Titanium Diboride

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Room temperature uniaxial and flexural strength, Weibull parameters, and fracture toughness of hot pressed $TiB_2$ are presented. Fracture toughness estimated from fractographic analysis was compared to the Single Edge Precracked Beam (SEPB) $K_{IC}$ measurements. Discrepancy between the estimated and measured values were rationalized by invoking the possibility of R-curve behavior. Fractographic analysis and the load displacement records of Vickers indented specimens point to the existence of rising R-curve behavior.

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# 1. INTRODUCTION

Titanium diboride ($TiB_2$) is used in a variety of wear applications, in evaporation boats, and Hall cell cathodes (Ferber, Becher, and Finch 1983). Because of its high hardness, low density, low Poisson's ratio, and high sonic velocity, $TiB_2$ ceramics have been recognized as an ideal candidate for armor ceramics (Viechnicki, Slavin, and Kliman 1991). Recently, $TiB_2$ has also been used as a second-phase particulate to form composites with other ceramics such as $\alpha$-SiC (McMurtry et al. 1987). The increasing use of $TiB_2$ ceramics in various applications, most of which require a significant level of structural integrity, demands reliable strength statistics and accurate fracture toughness data as critical design parameters. While data exists on the modulus of rupture (MOR) strength (Tracy, Slavin, and Viechnicki 1988; Katz et al. 1991) and some data is available on the fracture toughness (Tracy, Slavin, and Viechnicki 1988), limited data exists on the uniaxial strength and associated Weibull statistics.

This report presents a detailed investigation of the room temperature tensile and flexural strength, failure statistics, and room temperature fracture toughness of hot pressed (HP) $TiB_2$. Strength limiting flaws based on fractographic observations were used to estimate toughness using linear elastic fracture mechanics (LEFM). These estimations were compared with the toughness values measured by the Single Edge Precracked Beam (SEPB) method (Nose and Fujii 1988). Unlike previous studies (Katz et al. 1993; Cho, Katz, and Bar-On 1995), which indicated a close agreement between the estimated and measured values, toughness estimates from fractography in this case significantly underestimated the SEPB fracture toughness. The discrepancy between estimated and measured toughness values for $TiB_2$ are rationalized in terms of R-curve behavior. Attempts using the indentation strength in bending (ISB) technique (Chantikul et al. 1981; Cook and Lawn 1983) to observe direct evidence for R-curve behavior are presented.

## 2. EXPERIMENTAL PROCEDURES

2.1 <u>Material.</u> The $TiB_2$ used in this study was Cercom[*] PAD $TiB_2$. This $TiB_2$ was HP into a 15.24 × 15.24 × 2.54-cm billet. Published mechanical and physical properties are summarized in Table 1. Sixty MOR specimens with nominal dimensions of 3 × 4 × 50 mm (B type) were prepared in accordance

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[*] Cercom, Inc., Vista, CA.

Table 1. Mechanical and Physical Properties of Hot Pressed $TiB_2$

| | Cercom, Inc. (1994) | Conzone, Blumenthal, and Varner (1995) | Tracy, Slavin, and Viechnicki (1988) | Katz et al. (1991) |
|---|---|---|---|---|
| Density, g/cm$^3$ | 4.48 | N/A | 4.51 | N/A |
| Average Grain Size, μm | 15 | 13 | 10 | 9 |
| MOR, MPa | 277 | N/A | 334 | 398 |
| Weibull Modulus | 11 | N/A | 29 | N/A |
| Elastic Modulus, GPa | 556 | 526 | 551 | 563 |
| Poisson's Ratio | 0.11 | N/A | 0.11 | 0.10 |
| Hardness, GPa | 26.5[a] | 18.2[b] | 23.0[a] | 26.0[c] |
| $K_{IC}$, MPa$\sqrt{m}$ | 6.95[d] | 6.98 ±0.25[e] | 6.69 ±0.19[f] | N/A |
| Manufacturer | Cercom | Cercom | Ceradyne[g] | Dow[h] |

[a] Knoop.
[b] Believed to be Vickers.
[c] Vickers.
[d] SENB.
[e] SEPB using through thickness saw cut as a precrack starter.
[f] Double torsion test.
[g] Ceradyne, Inc., Santa Ana, CA.
[h] Dow Chemical Co., Midland, MI.

with the standard procedures (Army Materials Tech. Lab 1983; American Society for Testing and Materials 1994). An additional set of seven MOR B-type specimens was prepared for the ISB tests. Thirty Cylindrical tensile specimens with nominal dimensions of 9.2 mm in diameter and 120 mm in length were prepared by grinding the specimens circumferentially while meeting the same surface finishing procedures and requirements (Army Materials Tech. Lab 1983; American Society for Testing and Materials 1994) as for the MOR specimens.

2.2 Uniaxial Test. Tensile tests were performed at room temperature using a self-aligning hydraulic testing apparatus developed by Baratta and Driscoll (1971) using a straight cylindrical specimen as described by Hermansson, Adlerborn, and Burstrom (1987). Each end of the tensile specimen is inserted into a 40-mm-deep hole in a steel piston and adhesively bonded in place with a high-strength epoxy.[*]

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[*] ARALDITE AV 118, Ciba Geigy Corp., East Lansing, MI.

The specimen-piston assembly is inserted into the pressure chamber of the hydraulic tester.[*] The pressure is applied and increased until the specimen is broken apart by the hydraulic pressure acting against the pistons. A detailed description of this test method may be found elsewhere (Hermansson, Adlerborn, and Burstrom 1987; Katz, Lucas, and Toutanji 1994).

2.3 Fractography and $K_{IC}$ Estimation. The fracture surfaces of the tensile specimens were examined by both optical and scanning electron microscopy (SEM). High magnification SEM fractography and elemental analysis were carried out using an electron microscope equipped with an energy dispersive x-ray analyzer to measure failure initiating flaws as well as to examine the nature of such strength limiting flaws. Fracture toughness was estimated for tensile specimens having internal failure initiating flaws with fully developed circular mirror, mist and hackle, with their plane normal to the tensile axis (Katz et al. 1993), using Sneddon's solution (Sneddon 1949):

$$K_{IC} = 2\sigma_C \left(a_C/\pi\right)^{1/2}.$$

Here $K_{IC}$ is the mode I fracture toughness, $\sigma_C$ is the fracture stress, and $a_C$ is one-half the largest linear dimension of the critical flaw size. Small amounts of eccentricity in loading were corrected using optical microscopy by a technique described in Lucas (1991).

2.4 MOR Tests and Weibull Analysis. Four point bend tests were carried out in accordance with Army Materials Technology Laboratory (1983) and American Society for Testing and Materials (1994) using a fully articulating fixture having 20-mm inner and 40-mm outer spans. A screw-driven 25-kN load capacity universal testing machine with a 5-kN load cell was used. All specimens were fractured at a displacement rate of 0.5 mm/min. Fractographic analysis of the MOR specimens will not be reported in this study. Weibull parameters were calculated using the two-parameter Weibull equation (Weibull 1951) by fitting the tensile and MOR tests data using the maximum likelihood method (Thomas, Bain, and Antle 1969).

2.5 SEPB $K_{IC}$ Measurement. Broken halves from the MOR tests were used for SEPB $K_{IC}$ (Nose and Fujii 1988) tests using the experimental procedures described in Quinn et al. (1992). Indent loads ranged

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[*] ASCERA Hydraulic Tensile Tester, Robertsfors, Sweden.

from 68.6 N to 98 N. Using bridge indentation (BI) spans of 3–5 mm, pop-in loads were between 9.6 kN to 13.7 kN. All specimens were indented using a Vickers indentor mounted in a screw-driven 5-kN capacity universal testing machine at a displacement rate of 0.1 mm/min. The indent crack was extended by the BI fixture using a 250-kN capacity servo hydraulic universal testing machine with a 25-kN load cell. The loading rate was 1 kN/s.

The specimens were fractured in the three-point bending using a semi-articulating fixture with a 16-mm span. The displacement rate was 0.5 mm/min. A 5-kN capacity universal testing machine with a 0.5-kN load cell was used. The fracture surfaces were photographed at magnifications of 20 to 25 using low angle incident light. Precrack lengths were measured directly from the photographs at three equidistant points across the specimen width (Quinn et al. 1992).

Fracture toughness was calculated from Srawley's stress intensity solution (Srawley 1976) using the average precrack length determined from the three measurements. If the difference between any two of the three crack length measurements was greater than 10% of the average crack length, the test was rejected (American Society for Testing and Materials 1995). The test was also considered invalid if the plane of the pop-in precrack deviated more than 5 from the perpendicular to the tensile axis (American Society for Testing and Materials 1995; Bar-On et al., to be published). Adherence to this requirement was checked by measuring the angles between the precrack lines and the loading line at all four faces of the specimen before or after the fracture (Bar-On et al., to be published).

2.6 <u>ISB Test</u>. ISB tests (Chantikul et al. 1981; Cook and Lawn 1983) were performed to investigate possible R-curve behavior in $TiB_2$. Initially, seven B-type MOR specimens were selected and prepared for the ISB tests. One $4 \times 50$-mm face of the specimen was polished manually using 45 to 1-mm diamond pastes. In order to determine the appropriate indent loads for the formation of an acceptable median crack pattern (Chantikul et al. 1981), Vickers indents were placed on a polished surface with indent loads of 9.8, 29.4, 49, 68.6, and 98 N. All indentations were done following the procedures described in the previous section except that displacement rates of 0.05 and 0.01 mm/min were used. The crack patterns were then optically examined and photographed immediately after the indentation at a magnification of 200.

Three Vickers indentations were placed on the polished face of the ISB specimens within the inner span portion (Cook and Lawn 1983). Identical loads of 19.6, 39.2, 68.6, or 98 N were used for each

specimen 5 mm apart from each other. All ISB specimens were fractured in four-point bending at a displacement rate of 0.5 mm/min using inner and outer spans of 20 and 40 mm, respectively. Load vs. displacement was recorded using an analog x-y recorder. All specimens were photographed immediately after fracture at a magnification of 200.

## 3. RESULTS AND DISCUSSION

3.1 Tensile and MOR Strength. Figure 1 shows the Weibull plots of uniaxial and MOR tests together with the characteristic strength, $\sigma_{CHAR}$, and the Weibull modulus, m. Characteristic tensile strength obtained from this study is approximately equal to the previous reported MOR value (Cercom, Inc. 1994) (see Table 1). MOR strength measured in the present study was about 25% higher than the value obtained from the uniaxial test. The Weibull modulus (m) obtained by uniaxial tests, on the other hand, was nearly twice the value obtained from the MOR tests. Most of the low-strength data points for the flexural tests were located outside the lower 90% confidence band (not shown in Figure 1).

Theoretically, the characteristic Weibull MOR strength can be predicted from the tensile Weibull parameters by equating the unit volume characteristic strength obtained for the tensile and flexural specimens (McLean and Hartsock 1989). A predicted value of 405 MPa was obtained, which was ~18% higher than the measured value. This disagreement and the deviation from the confidence limit might be an indicative that more than one flaw population is present in the material. The different flaw populations might respond differently to the shear loading encountered in four-point bending and to the pure tensile mode (mode I).

3.2 SEPB Fracture Toughness. SEPB fracture toughness data for the $TiB_2$ material is listed in Table 1 along with the values measured by different methods taken from the literature (Tracy, Slavin, and Viechnicki 1988; Katz et al. 1991; Cercom, Inc. 1994; Conzone, Blumenthal, and Varner 1995). The mean SEPB $K_{IC}$ value obtained from the six valid SEPB $K_{IC}$ tests was $6.25 \pm 0.25$ MPa$\sqrt{m}$ .The HP $TiB_2$'s used in references (Cercom, Inc. 1994; Conzone, Blumenthal, and Varner 1995) and in this study are believed to be similar material (Shih 1995). Cercom, Inc. (1994) used the SENB method to measure the toughness, while Conzone, Blumenthal, and Varner 1995) and the present work used the SEPB method. The SENB method typically results in higher toughness values as compared with the SEPB method because of the blunt crack tip. The difference between Conzone, Blumenthal, and Varner (1995) and this work is the choice of the precrack starter. This study used Vickers indentation as a precrack starter, whereas Conzone, Blumenthal, and Varner (1995) used a through thickness saw cut as a precrack
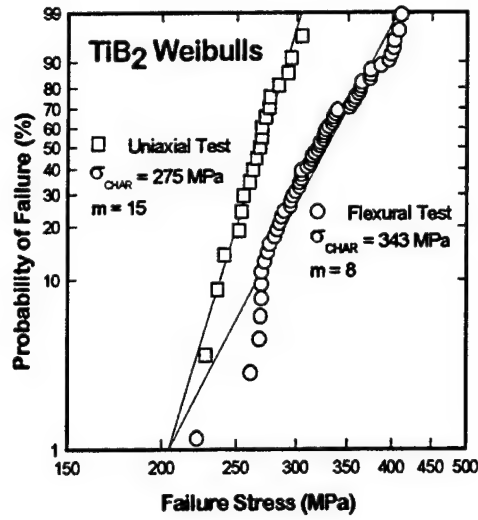
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Figure 1. Weibull plots of the uniaxial and MOR tests generated by the maximum likelihood method.

starter. The authors are aware of the residual stress effects in measured SEPB toughness values (i.e., higher indent loads tend to result in lower measured SEPB toughness). A previous study on AIN (Bar-On et al., to be published) indicated, however, these effects were negligible if indent loads were about 98 N or less. In this present work, three, one, and two specimens were indented using 98, 78.4, and 68.6 N indent loads respectively and their respective toughness values were $6.20 \pm 0.17$, $6.37$, and $6.25 \pm 0.23$. These values are within the experimental scatter when compared to the combined mean value of $6.25 \pm 0.25$ MPa$\sqrt{m}$ .

Figure 2 shows the SEPB $K_{IC}$ values grouped by two requirements for a valid test. No increase trend in measured $K_{IC}$ value is observed up to $\Delta a_{max}/a_{avg}$ of 12%. A $K_{IC}$ value of nearly 7 MPa$\sqrt{m}$ is obtained for a $\Delta a_{max}/a_{avg}$ ratio of 36%. But this data point was obtained from a specimen containing a nonideal precrack plane whose deflection angles are 2, 5, 8, and 10 from the loading plane measured at the four sides of the specimen. In the present work, one of the valid SEPB $K_{IC}$ test requirements was that the deflection angles be 5 or less. Conzone, Blumenthal, and Varner (1995), however, followed the requirement specified in JIS R 1607 (Japanese Standard Association 1990), which allows a maximum deflection of 10. In addition, Conzone, Blumenthal, and Varner (1995) used a span-to-width ratio of nearly 5, while the present work used 4. Both studies used Srawley's stress intensity solution (Srawley
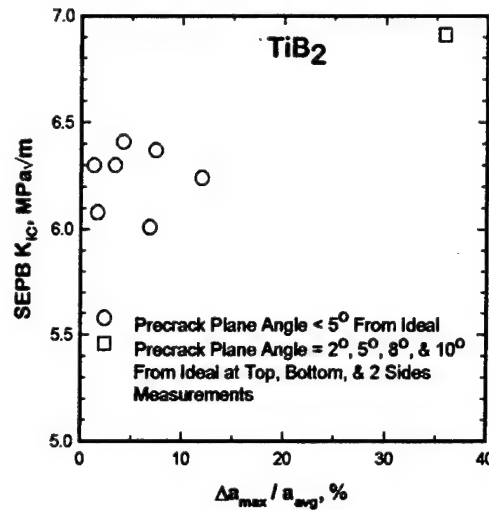
Figure 2. SEPB $K_{IC}$ values are grouped with two requirements for a valid test.

1976) to calculate $K_{IC}$. By taking these differences in span-to-width ratio and deflection angles into account, the difference in measured SEPB $K_{IC}$ values between this study and Conzone, Blumenthal, and Varner (1995) can be accounted for.

3.3 Fracture Toughness Estimation From Fractography. Four tensile specimens appeared to meet the criteria for estimation of $K_{IC}$. An estimated toughness value of $4.40 \pm 0.36$ MPa$\sqrt{m}$ was obtained, which significantly underestimates the measured SEPB $K_{IC}$ value of $6.25 \pm 0.25$ MPa$\sqrt{m}$. It is reasonable that the difference in fracture toughness encountered between large artificially induced flaws, such as in an SEPB test, and the small naturally occurring flaws used in fractographic estimation, would be very sensitive to R-curve behavior. Swab and Quinn (1994) point out that one cause of observing critical flaws significantly smaller than those predicted from macroscopic fracture toughness tests is the presence of R-curve behavior. The ranges of flaws measured on TiB$_2$ tensile fracture surfaces was 170 ~ 280 mm. The average flaw size calculated using the average tensile strength of 280 MPa and the measured SEPB $K_{IC}$ of 6.25 MPa$\sqrt{m}$ is ~390 mm. The observed flaw sizes were significantly smaller than the average anticipated flaw size.

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An alternative mechanism may be stable crack growth. Although no analytical crack stability solution exists for the tensile loading of small naturally occurring flaws, it is reasonable to assume that small cracks will be more susceptible to stable crack growth as evidenced in an indentation study (Chantikul et al. 1981). In this case, the fracture initiating flaws may not be the same as the critical flaws. Whether associated with R-curve behavior, or crack stability (with flat R-curve behavior), the size of the fracture initiating flaw will not be that of the critical flaw for fracture mechanic calculation purposes. An interesting example of this was encountered in one $TiB_2$ specimen examined in this study. Figure 3(a) shows the large grain at the center of the "pseudo-mirror." Figure 3(b) shows that this grain itself has a true fracture mirror with a small (~20 μm) particle at the center of the mirror. This inclusion showed traces of W, Co, Fe, and Ni, which are all consistent with a fragment of WC grinding media. It is possible that this inclusion was the initiating flaw, but it is much too small to be the critical flaw. Similarly, the large grain is too small to be the critical flaw predicted by fracture mechanics based on the conventional large flaw toughness measurement. It is evident that care should be taken not to use the terms "fracture initiating flaw" and "critical flaws" interchangeably.
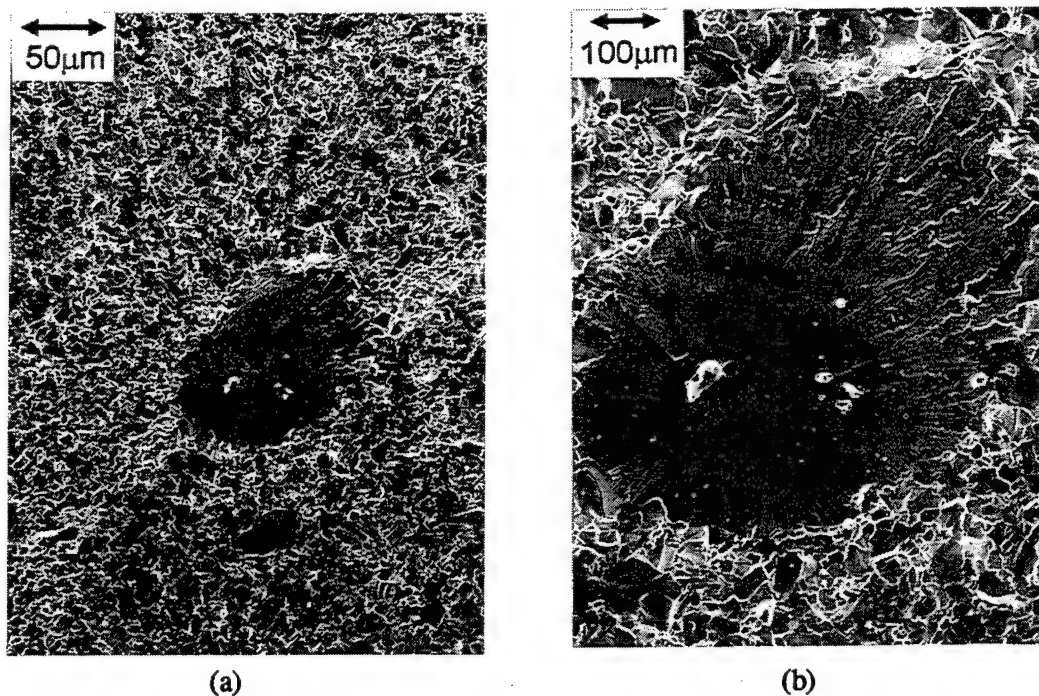


(a)                                                    (b)

Figure 3. Fracture origin of $TiB_2$ specimen shows (a) a large grain at the center of the "pseudo mirror" and (b) a small particle within the large grain.

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3.4 <u>ISB Test</u>. To document rising R-curve behavior in TiB$_2$, ISB tests were attempted. There are two requirements (Chantikul et al. 1981) for a valid indentation study of the median crack system, which consists of two half-penny shaped cracks (Lawn, Anstis, and Marshall 1980): (1) A well-defined symmetrical indentation impression and (2) four cracks of similar lengths emanating from the corners of the indent without crack branching. Requirement (1) was only satisfied for the Vickers indentations with indent loads of 19.6 and 39.2 N. Impressions with indent loads of 68.6 N and above had one or more crushed corners or sides instead of having well-defined symmetrical Vickers impressions. None of the indentations fulfilled requirement (2). Instead of having four cracks of similar lengths initiating from the corners, severe crack branching and cracks occurring from the sides were observed.

ISB tests were attempted to compare our results with the indentation strength toughness (IS K$_c$) (Chantikul et al. 1981) values reported by Conzone, Blumenthal, and Varner (1995). For the specimens with indent loads of 19.6 and 39.2 N, however, failures did not initiate from the Vickers created flaws. Failure locations were within the 20-mm inner span of the four-point loading. This indicates that processing or machining related flaws were larger than the controlled flaws created by Vickers indentation. No difference in Vickers impressions was observed from the specimens indented at a displacement rate of 0.01 and 0.05 mm/min. It was impossible to obtain a well-defined impression even at the lower loading rate. For the specimens with 68.6 and 98 N indent loads, cracks did initiate from the controlled flaws created by the Vickers indentation. Although both the IS K$_c$ or the ISB test do not require a direct crack length measurement, the two requirements described previously need to be met. Furthermore, the empirical stress intensity expression (Chantikul et al. 1981) and the two calibration constants, one associated with the Vickers induced residual stress field and the other associated with far field applied stress field, assume a well-developed median crack system.

While these difficulties did not permit the calculation of stress intensity values, it was observed that the load displacement records showed a distinct nonlinearity prior to fracture. This feature could be an indication of extension and arrest of a flaw. Such behavior would be an indication of increasing crack growth resistance.

## 4. SUMMARY

Uniaxial strength, flexural strength, and fracture toughness were measured for HP TiB$_2$. The comparison together with fractography and the load displacement records of indented specimens points to the existence of rising R-curve behavior.

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